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# An Investigation into the Recycling of Ti-6Al-4V Powder Used Within SLM to Improve Sustainability

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**Abstract** *This paper considers the steps to be taken in investigating the effects of recycling Ti-6Al-4V powders for Selective Laser Melting (SLM). A research methodology is presented that shall allow for investigation in to the change in powder characteristics through repeated use, and will provide the basis for eventual determination of any effect that these changes have on the chemical, mechanical and metallurgical properties of laser melted parts. An in depth understanding of the link between powder and part characteristics is essential for SLM, and measuring powder characteristics after building production parts may prove to be an important marker for part quality. Initial powder characterisation testing was conducted on powder that had been recycled up to five times. The results showed a trend towards increased Particle Size Diameter (PSD) through repeated recycling, with a reduction in the number of fine particles. Additionally, a roughening of the powder surface was observed, with a reduction in the sphericity of powder particles. Chemical composition tests on the powder to determine changes in interstitial oxygen and nitrogen showed negligible change through repeated recycling.*

## 1. Introduction

Titanium and its alloys are relatively expensive to produce due to difficulties in refining. As a result, "near-net-shape" manufacturing techniques are desirable for reducing the material waste and stages of component manufacture. "Near-net-shape" manufacturing provides the means to improve the sustainability of manufacturing processes when compared to traditional subtractive technologies [1]. Many CNC machining processes use billet material, with billet part ratios as high as 20:1 being possible. This means that for 1 kg of part produced, there can be 19 kg of material that enters the waste stream [2]. A reduction in the material required to produce parts clearly improves the sustainability of manufacture due to less waste being produced and less raw material production required.

Selective Laser Melting (SLM) is one such “near-net-shape” manufacturing technique that shows great potential for the manufacture of Ti-6Al-4V components for medical, dental, automotive and aerospace industries. Production of suitable powders for SLM involves high costs due to restricted volume capabilities as a result of low process yields in Plasma Atomisation (PA) [3]. The desire to utilise a sustainable manufacturing process and to keep manufacturing costs of components as low as possible drives the requirement for recycling of Ti-6Al-4V powder that is un-used within the SLM process.

The recycling of Ti-6Al-4V powders has been shown to affect the powder Particle Size Distribution (PSD) and morphology, which may affect the density, hardness and mechanical strength of produced parts [4]. It has also been shown during investigation into SLM of steel powders, that changes in powder characteristics change the powder bed packing density [5, 6, 7]. In addition to changes in size and shape, the high reactivity of Ti-6Al-4V powder provides the potential for the absorption of interstitial elements carbon, hydrogen, oxygen and nitrogen into the powder [8, 9]. Industrial standards stipulate limits of interstitial elements and so the rate of absorption of these interstitial elements in to both powder and laser melted parts must be understood.

This paper details the steps that shall be taken in conducting a systematic study in to the effects of recycling of Ti-6Al-4V powders used within SLM. This study concentrates on determining the change in powder characteristics in terms of PSD, morphology and chemical composition. The point at which powder has reached its useable life shall be determined for a variety of relevant industrial standards.

## 2. Research Aims & Objectives

Ti-6Al-4V parts produced through SLM must conform to the relevant standard stipulated by the industry into which the parts shall be in service. These standards specify allowable limits of chemical composition and mechanical properties. The recycling of titanium powders used within SLM is believed to change the powder properties in terms of chemical composition, particle size distribution and morphology, which shall affect the flowability of powder and packing density on the build plate. In addition to this, changes in chemical composition of the powder may be transferred in to the parts produced within the SLM process.

As such, an understanding of the way in which powder characteristics change through repeated recycling is fundamental in the development of SLM for titanium alloys. The link between powder characteristics and the effect that this shall have on final part quality shall be investigated thoroughly in the future. To improve process sustainability, the powder must be recycled as many times as possible, whilst still maintaining desirable composition characteristics that are capable of producing parts that satisfy industrial standards. This paper shall focus on the work completed in characterising powders at various stages of recycling.

## 3. Research Methodology

The initial steps in this study involved determining experimentally the effect that repeated recycling had on the powder used within the process. As such, an investigation was conducted to control the recycling of powder used within SLM across five repeated builds. A large volume of Extra Low Interstitial (ELI) Ti-6Al-4V (Grade 23 ASTM F136-13) powder produced from one manufacturing batch was collected and used as the feed stock for the investigation.

The SLM machine was loaded with 40 kg of virgin powder. Careful calculations were made to estimate the quantity of powder required to produce five consecutive builds, whilst accounting for material removal in solid parts and powder lost to sieving, filtering and overflow. The build shown in Figure 1 was produced. Upon completion of the build, the remaining powder was dosed into the machine layer by layer to ensure that it was exposed to the same thermal and mechanical work history as the powder used within laser melting, until the hopper was empty.

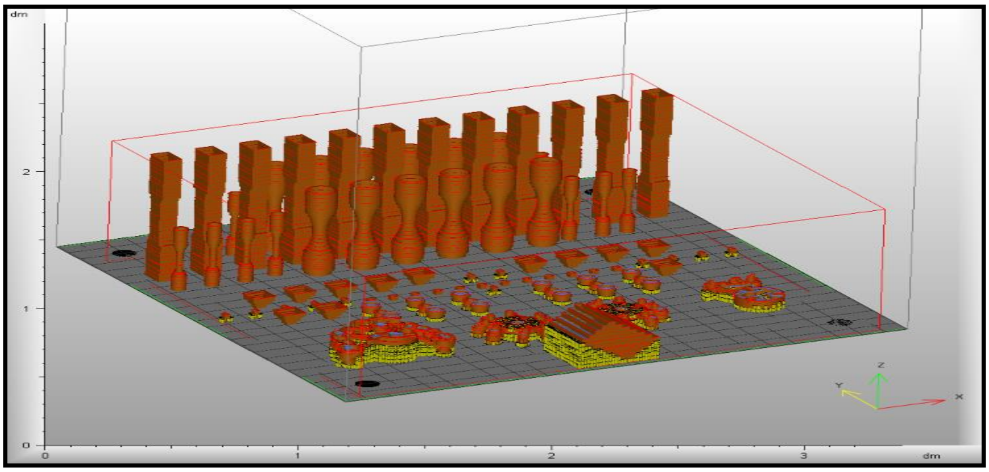


*Figure 1 - CAD Model of Build for Testing*

Upon completion of the build and dosing, the build plate was removed, and all powder swept into the overflow containers for sieving and re-introduction to the powder hopper. The build plate was sent for heat treatment. The powder in the hopper had now been used once previously and was used to produce another identical build. This process continued until five identical builds had been produced.

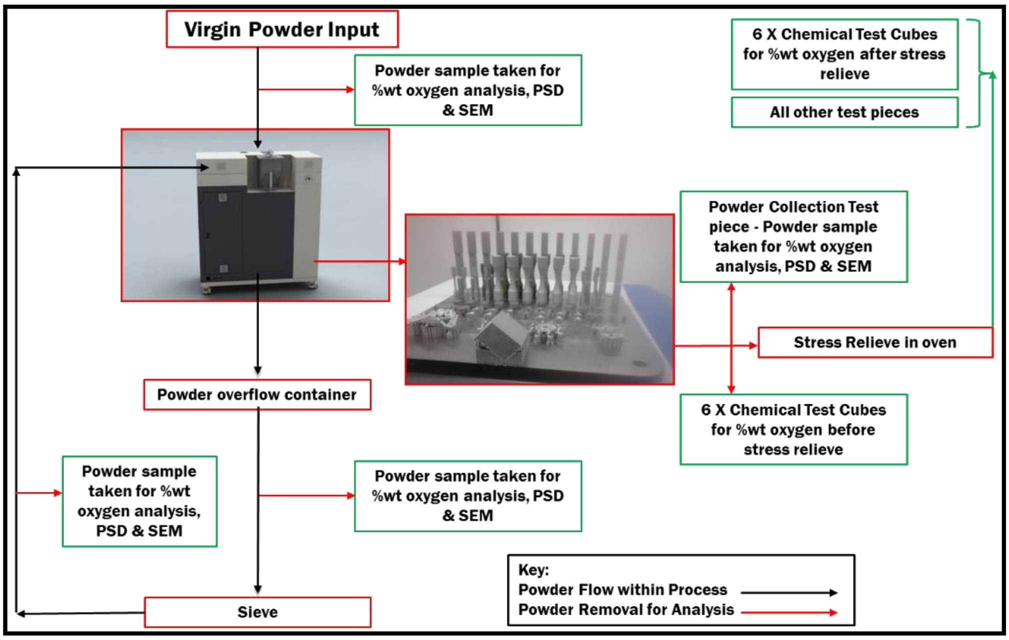


*Figure 2 - Diagram of Powder Flow and Sampling through Testing*

This testing aimed to determine the change in powder characteristics throughout the SLM process. This included analysis of virgin powder samples, samples from within the build chamber (denoted Build 'X' C) in the gaps between laser melted parts and after each sieving process (denoted Build 'X' PS). Powder sampling locations are shown in Figure 2, along with the powder flow routes and removal of parts. The build plate contained a series of test pieces that were intended to determine the change in mechanical, metallurgical and chemical properties of produced components through repeated recycling of titanium powder and shall be reported on at a later date.

The machine parameters used within testing were maintained throughout all builds and are shown in Table 1. Layer thickness was maintained at 40 µm, with a total of 2567 layers to attain the total build height of 102.68 mm. The machine in question had a maximum limit of 1000 ppm (0.1%) oxygen concentration. Above this limit, no laser melting would occur. It can be seen in Figure 3, that over the course of the first 30 minutes, the oxygen concentration within the bottom chamber of the machine dropped to around 0. This occurred at around layer 30. With the layer height of 40 µm, 30 layers would amount to a build height of 1.2 mm. With a minimum support height of 3 mm, there should have been very little oxygen remaining within the build chamber when the actual parts began being built.

*Table 1 - Processing conditions*

| | Volume border | Volume area | Supports |
|---|---|---|---|
| **Power (W)** | 200 | 200 | 170 |
| **Point Distance (µm)** | 50 | 70 | 60 |
| **Exposure time (µs)** | 35 | 65 | 50 |
| **Scan strategy** | - | Meander hatch | - |

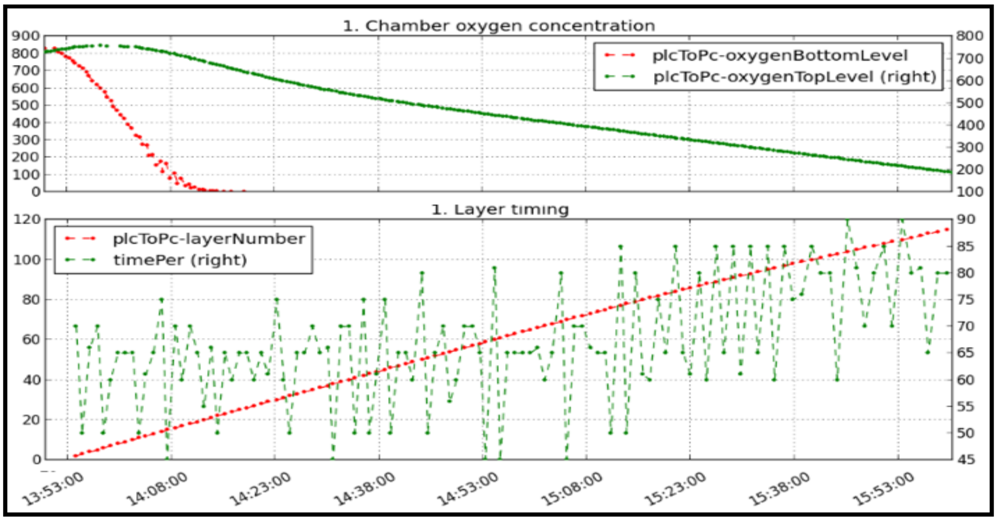


*Figure 3 - Data logged over the First Two Hours of Production*

Several methods for characterising powders were used. These being:-

- Powder Particle Size Distribution (PSD) through the use of a Malvern Mastersizer. This machine used laser diffraction to determine particle sizes held within a wet dispersion solution.

- Particle morphology and surface microstructure, viewed under Scanning Electron Microscope (SEM). This provided the means for determination of change in particle shape with repeated recycling and provide additional confidence in results of PSD obtained through laser diffraction.
- Chemical analysis conducted as per ASTM E1409 – Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique.

## 4. Results

### 4.1. Powder Characterisation

#### 4.1.1. PSD Analysis

An important consideration when determining the change in powder characteristics was to determine the change in PSD for the samples collated. The analysis was conducted using a Malvern Mastersizer 3000 Laser Diffraction Particle Size Analyser. Each sample was analysed five times, with the average of the five runs being displayed. The change in PSD can be seen in Figure 4 and Figure 5. Through repeated recycling, there was a reduction in the number of fine particles (<15 µm) within each sample analysed and an increase in the number of larger particles (>45 µm). Overall, there was an increase in Dx(10), Dx(50) and Dx(90) diameter values through repeated recycling, as shown in Figure 6.

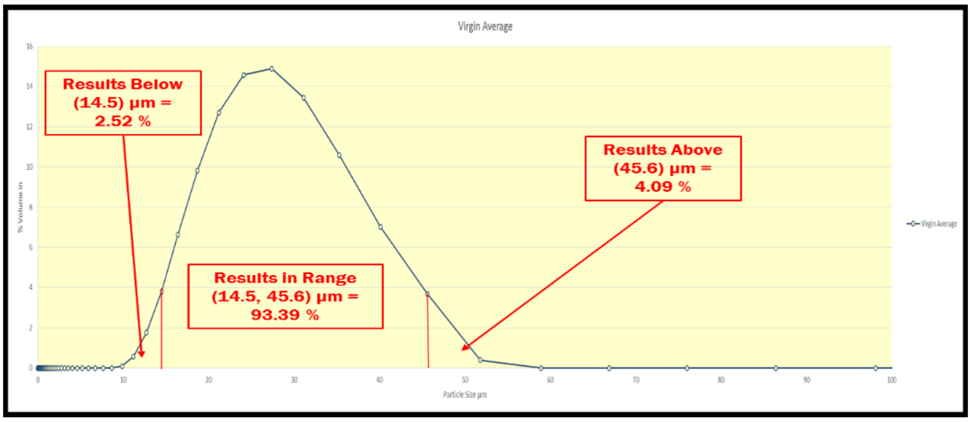


*Figure 4 - Average PSD Measurement for Virgin Powder*

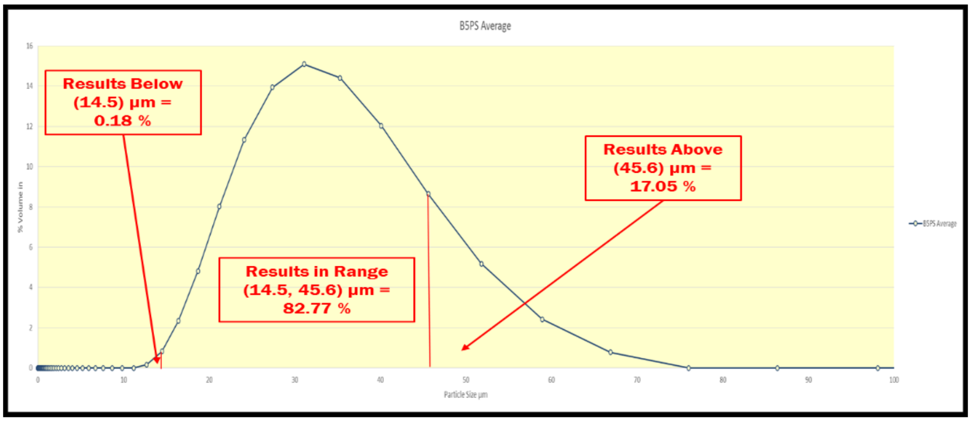


*Figure 5 - Average PSD Measurement for Build 5 Post Sieve*

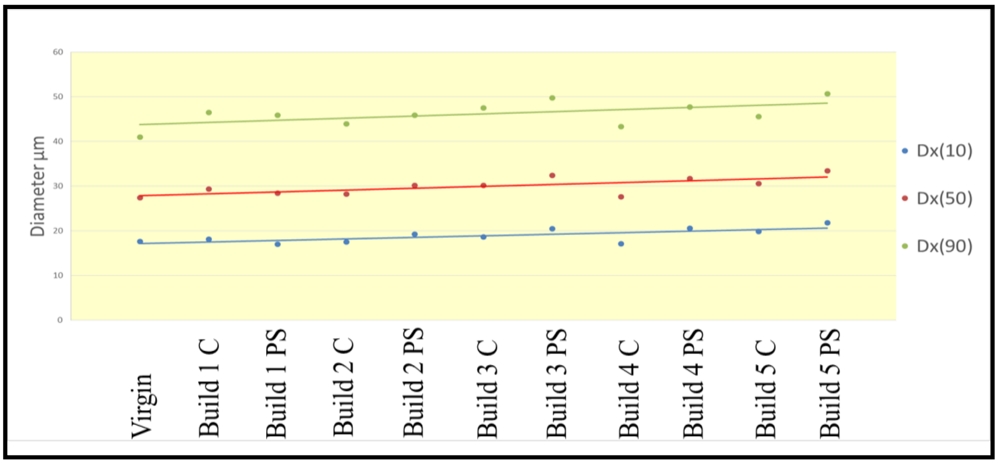


*Figure 6 - Dx(10), Dx(50) & Dx(90) Results for all Samples*

### 4.1.2. SEM Analysis

Virgin powder particles showed a high level of sphericity with the presence of satellite particles attached as shown in Figures 7 A & B. In all cases analysed in detail, where satellite particles were observed, there was the presence of extreme fine and nano scale particles attached in the joins, as shown in Figure 7 C. These extreme fine micro and nano scale particles, were not observed at any other point within the samples analysed. Virgin powder exhibited a mixture of coarse acicular martensitic alpha and large equiaxed structures, with a relatively smooth surface which is again shown in Figure 7 B. Even after only being used in one build cycle, there was a clear roughening of the surface. This contrast in surface can be viewed in Figure 7 B & D and Figure 8 C.

Consistently within the samples analysed, there was the presence of larger particles that were outside the intended range of particle size. These particles were up to 110 µm in diameter. Figure 8 A and B show a particle collected from the fourth build within the chamber, meaning that the powder had been sieved three times previously. The large particles consistently exhibited an extremely fine acicular martensitic alpha structure, suggesting extremely rapid cooling from the beta domain of the phase diagram. The high level of sphericity of these particles rules out partial sintering during the SLM process, which is shown in Figure 8 D. Such large particles were also observed within the virgin powder samples, meaning that they resulted from improper sieving of the manufacturers powder following their plasma atomization production process. These particles, however, did not exhibit the same extremely fine acicular martensitic alpha structure.

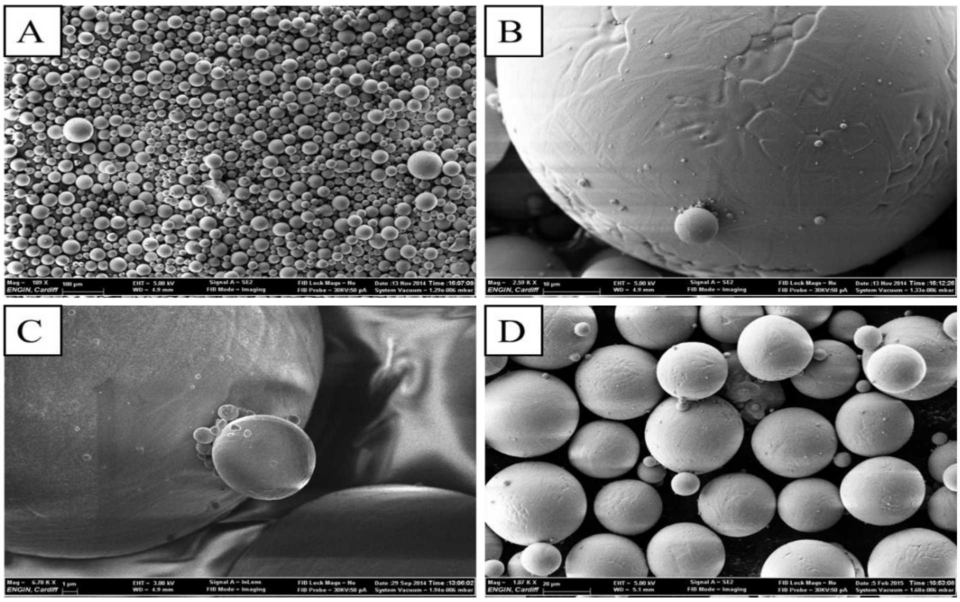


*Figure 7 - SEM Images of A. Virgin Powder Low Magnification. B. Virgin Powder Showing Satellite Particle Attachment. C. Virgin Powder Fine Patricles Attached in Join of Satellite Particle. D. Once Used Powder Showing Roughening of Surface.*

### 4.1.3. Chemical Analysis

Chemical analysis was conducted on the feed powder for each build. This meant that virgin powder, and powder that had been collected, and sieved after each build had a sample removed for analysis before being re-introduced to the powder hopper. The results are shown in Figure 9. The results show no significant trend in the change of %wt for both O and N through the recycling of the powders. It was clear, however, that the ELI Ti-6Al-4V powder supplied contained an oxygen content that was extremely close to the allowable limit of 0.13%, as described within ASTM F136-13. This suggested that even a low amount of interstitial elemental absorption into molten material within the SLM process would produce parts that do not conform to the chemical requirements stipulated.

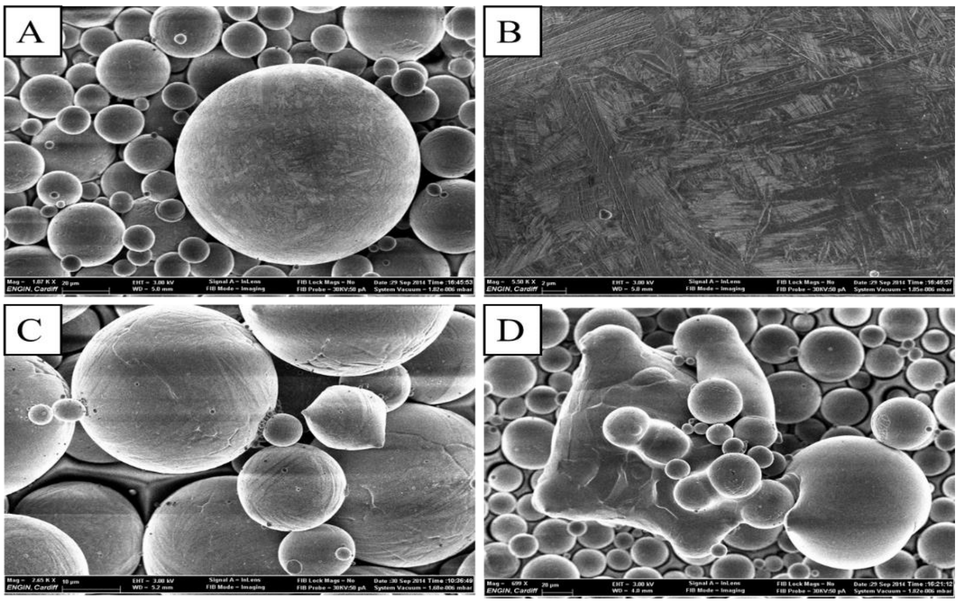


*Figure 8 - SEM Images of A. Powder from Fourth Build Showing Large Particle. B. Magnified Image of A Showing Fine Acicular Martensitic alpha structure. C. Powder taken from the chamber after the fifth build D. Powder from Third Build Showing Partial Sintering.*

### 4.2. The Effect on Produced Parts

Currently, the only testing on produced parts has been chemical analysis of test cubes. The results of chemical composition are shown in Figure 9. The chemical test cubes were removed from the build plate after SLM and had not been heat treated. The results indicate the level of oxygen and nitrogen absorbed into the part as a result of the SLM process. Additional chemical test cubes are to be heat treated to investigate interstitial element absorption during their heat treatment processes.

A comparison of the chemical composition of the powder used, and that of the parts produced from said powder, shows that even under vacuum conditions coupled with the flow of the inert gas argon across the build plate in process, there shall still be absorption of interstitial elements into the melt pool. There appears to be no increase in oxygen and nitrogen within Ti-6Al-4V powders for low numbers of recycling. It is not possible to say at this stage whether this would be the case for higher numbers of recycling.

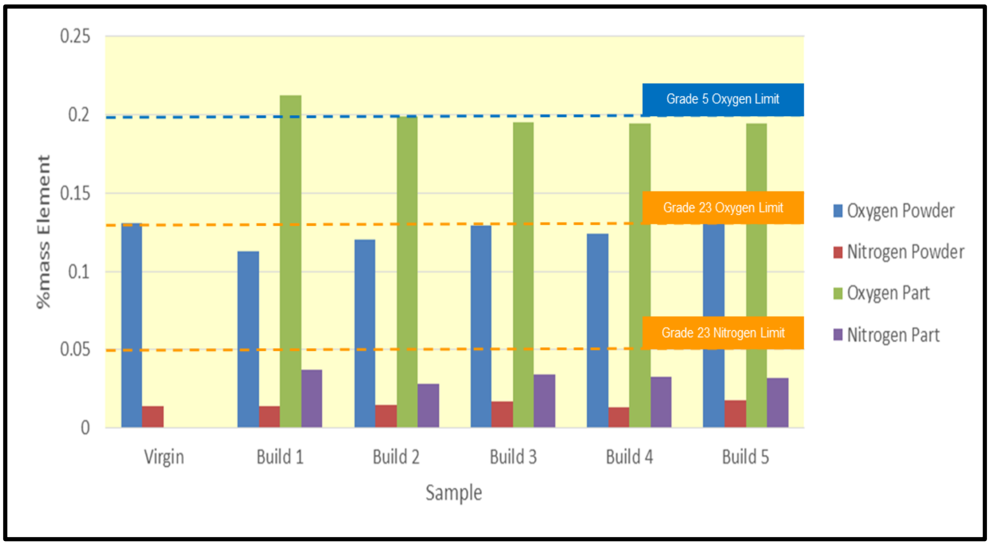


*Figure 9- LECO Analysis Results for Powder and lParts at Various Stages of Recycling*

## 5. Conclusions

Upon completion of the investigation in to the effects on powder characteristics through repeated recycling within SLM, the main findings were:

- The number of fine particles (<15 µm in diameter) as a percentage of the population reduces significantly while the number of larger particles (>45 µm in diameter) increases.
- Overall, there was a trend towards increasing diameter values of Dx(10), Dx(50) and Dx(90) through repeated recycling. Powder surfaces became rougher and less spherical through repeated recycling.

- There was no significant trend observed for change in chemical composition of the powders through repeated recycling. Ti-6Al-4V powders have been shown to have an extremely stable protective oxide layer comprised of $TiO_2$ and $Al_2O_3$, which could prevent additional interstitial element absorption in powder state.
- Even with negligible oxygen concentration within the build chamber and the flow of argon gas across the build plate, there was significant absorption of oxygen in to the laser melted parts.
- Parts produced through SLM using Grade 23 ELI Ti-6Al-4V powders did not conform to the chemical composition limit of 0.13 %wt, due to interstitial element absorption. This absorption pushed the oxygen %wt of Grade 23 ELI Ti-6Al-4V powder precariously close to the %wt oxygen limit (0.2%) of grade 5 Ti-6Al-4V (ASTM F1472-14) for all test cubes except those from Build 1, which were above the limit.

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